

# *Protective Clothing from Nanotube Based Fabrics*

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Fire Research Division

National Institute of Standards and Technology

# FIRE FIGHTER PROTECTIVE CLOTHING



**Compression Points:**

**SCBA Straps,  
Shoulders**

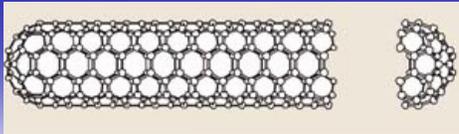
**Elbows, Wrists, Hands**

**Waist, Thigh, Knees**

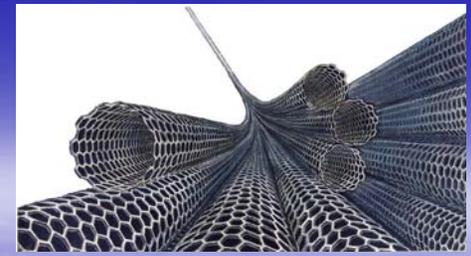


**Lower Insulating Capacity.**

**Increases Burn Injury Risk**



## Carbon nanotubes (CNTs)

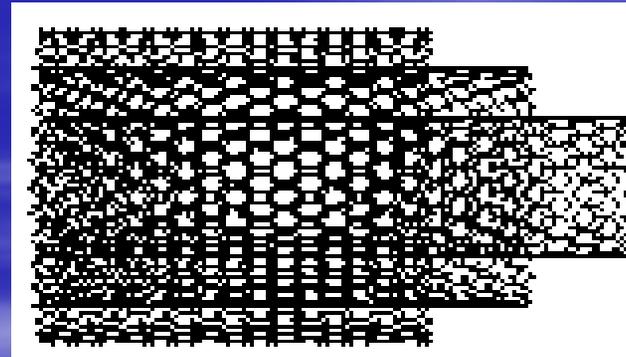
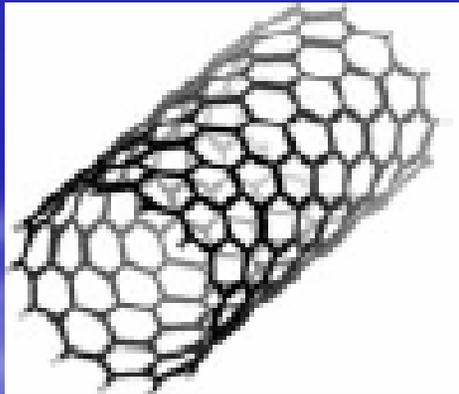


Discovered in 1993  
Diameter: 0.4 to 3 nm

Discovered in 1991  
Diameter: 4 to 50 nm

Single-wall carbon nanotube (SWNT)

Multiwall carbon nanotube (MWNT)



<u>Material</u>	<u>Conductivity (W/mK)</u>
Carbon nanotube	37,000 – 2,500 (calc)
Graphite	100-200
Polymers	0.01 – 20

# Comparison of Pure SWNT and PAN/ SWNT Nanocomposite :

## SWNT FILM

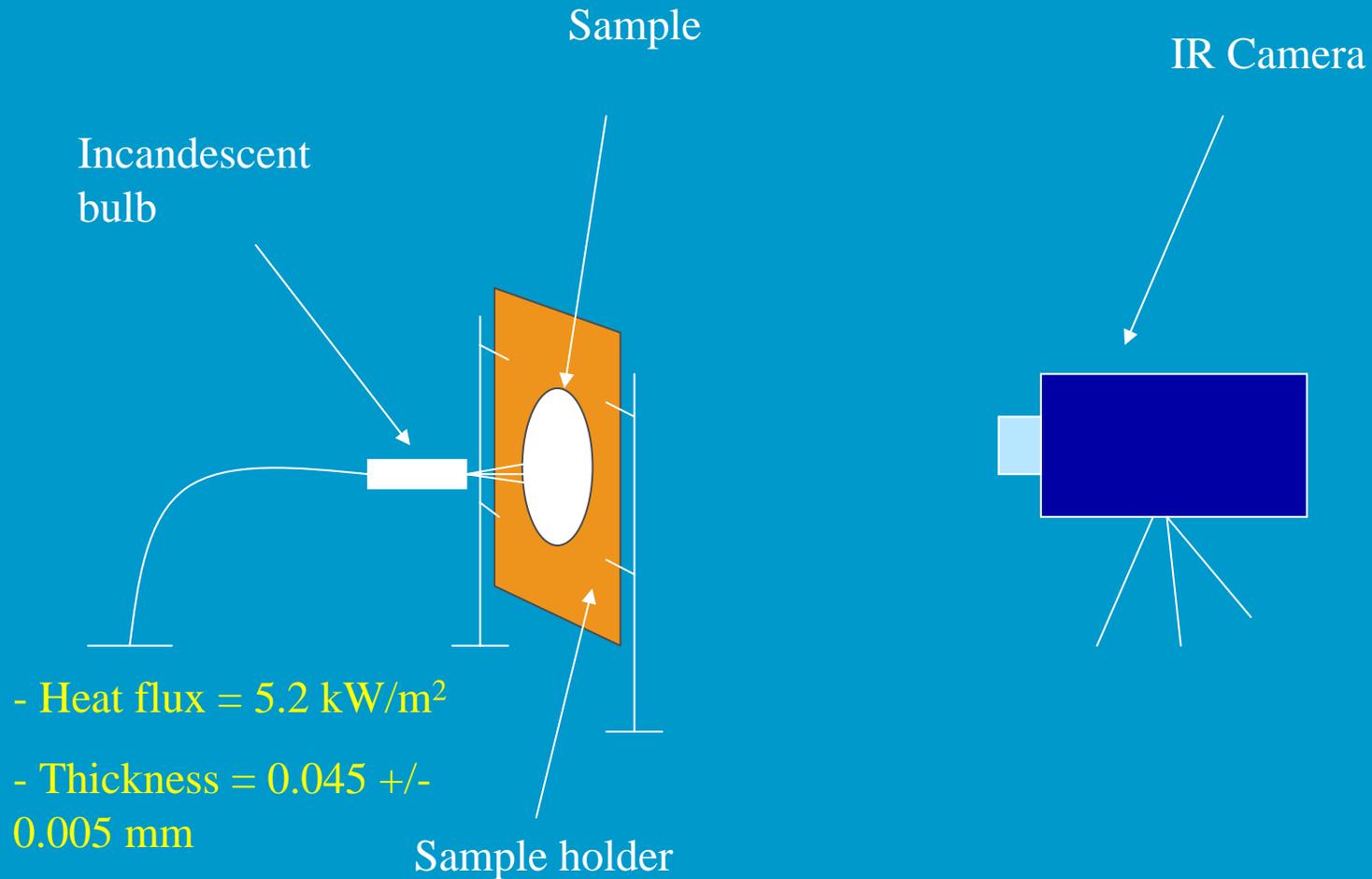
- SWNTs dispersed nitric acid refluxed at  $\sim 130^{\circ}\text{C}$ .
- centrifuged and washed with water
- sonication and filtration
- SWNT film dried under vacuum at  $100^{\circ}\text{C}$ .

## PAN/ SWNT NANOCOMPOSITE FILM

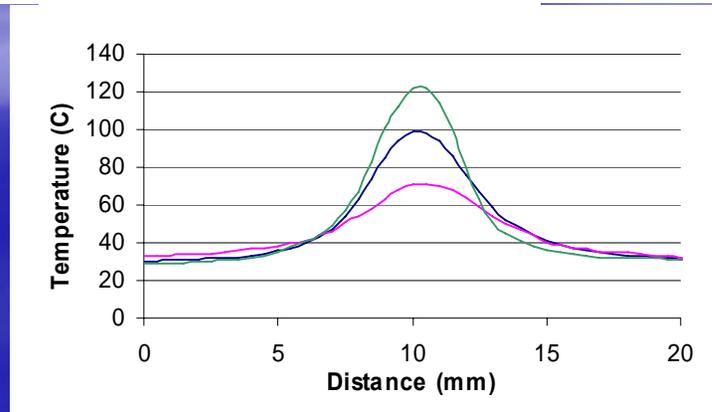
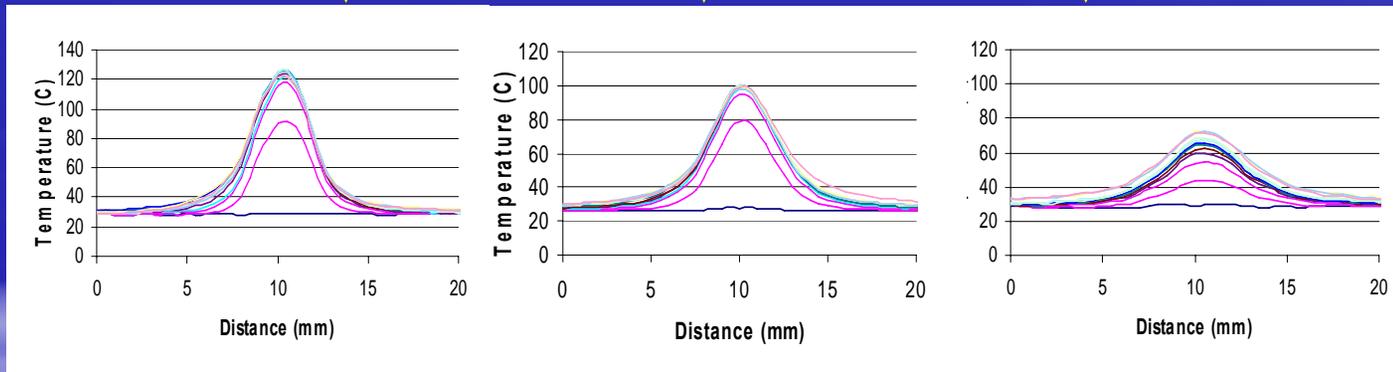
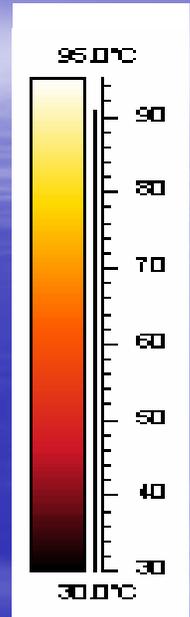
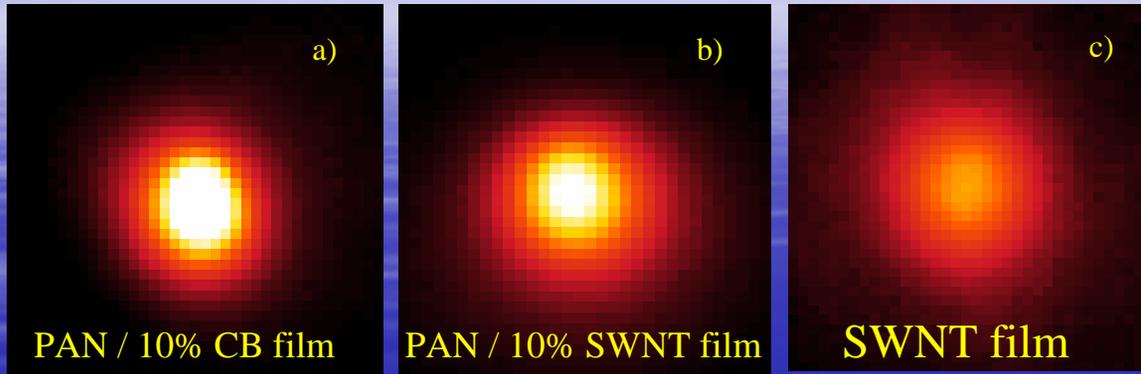
- SWNT mixed with excess solvent (DMF)
- and sonicated at room temperature
- PAN added to the SWNT dispersion
- and dissolved ( PAN: SWNT = 90: 10).
- The dispersion was poured into a glass mould and the solvent was slowly evaporated over a hot plate.
- Films courtesy of Satish Kumar, GA Tech



# IR Camera Apparatus

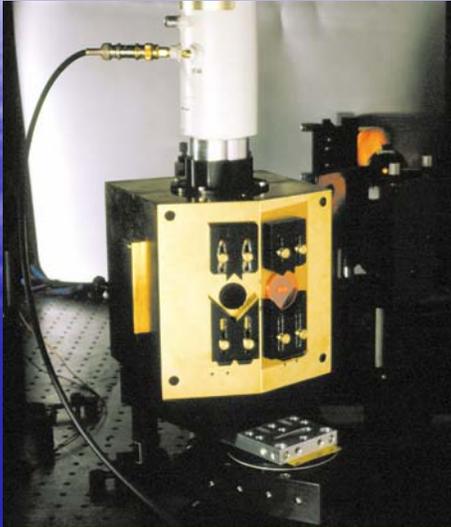


# IR Camera Images



- Heat flux =  $5.2 \text{ kW/m}^2$
- Thickness =  $0.045 \pm 0.005 \text{ mm}$

# Integrating Sphere for Specular and Diffuse Samples

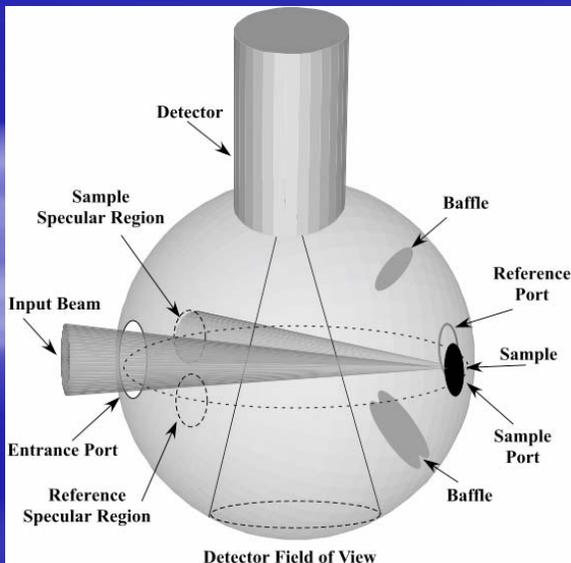


## Specifications

- $\lambda$  range: 1.0 - 18  $\mu\text{m}$
- 6 inch diameter
- gold-electroplated plasma-sprayed metal coating
- MCT detector w/ concentrator optics
- baffling in sphere
- $8^\circ$  incidence angle

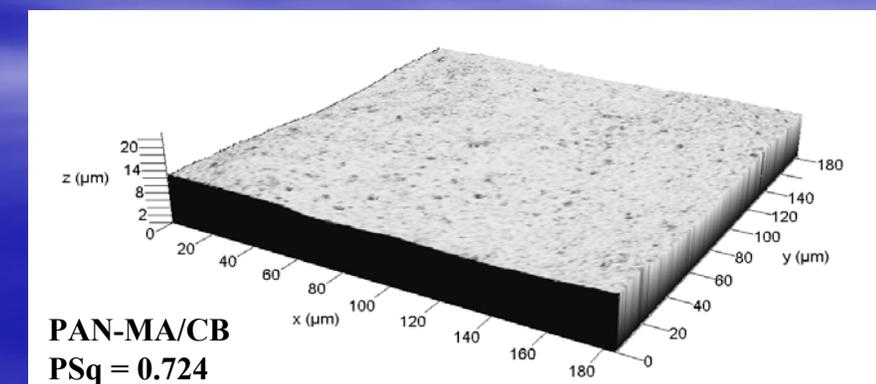
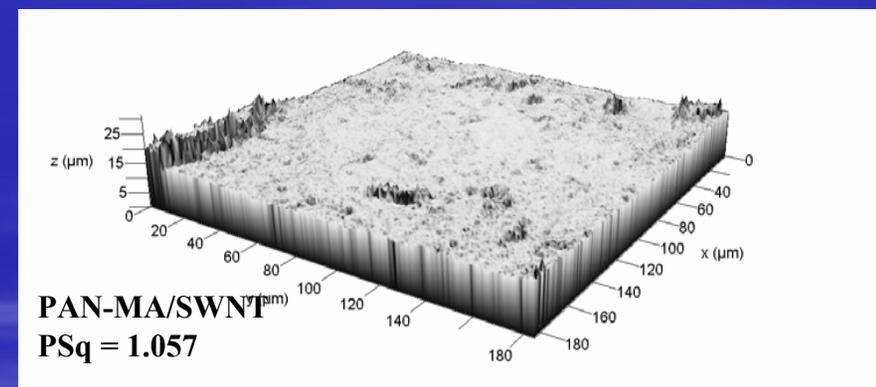
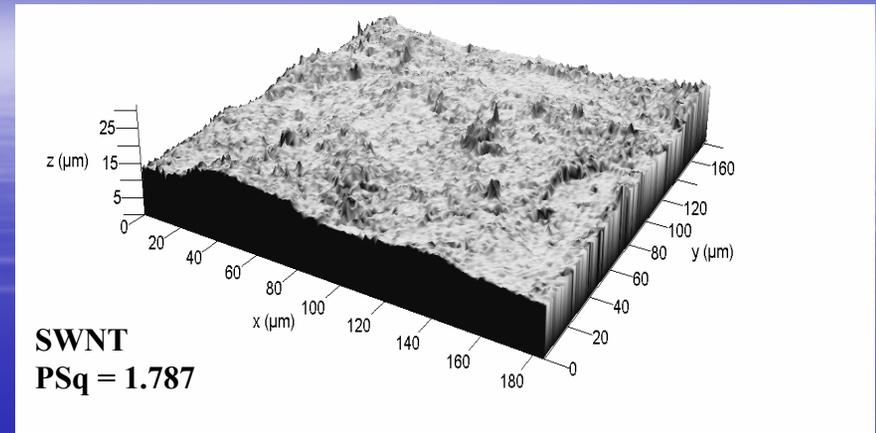
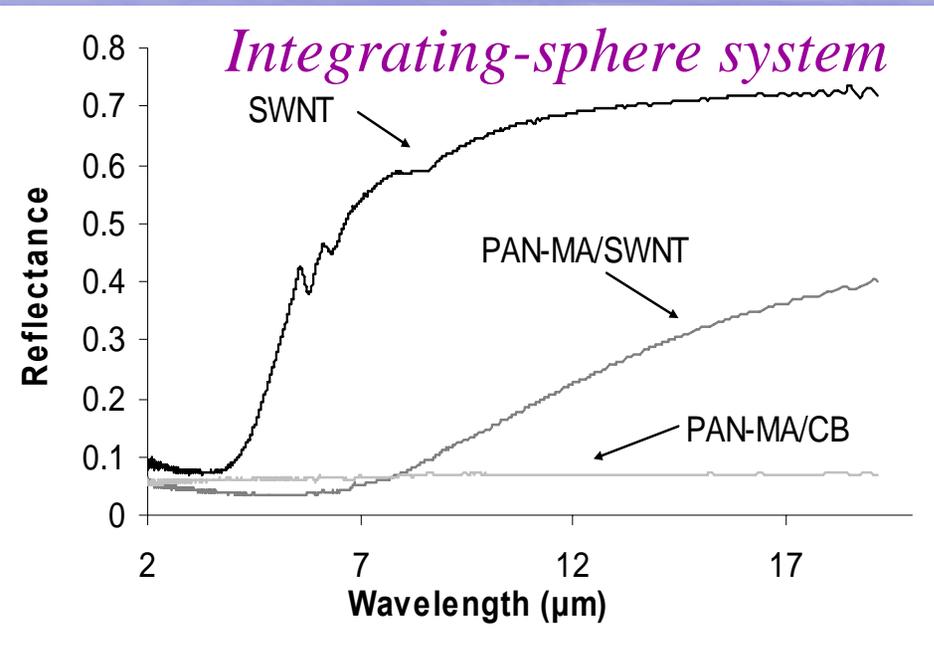
## Capabilities

- Reflectance, Transmittance & Absorptance
- absolute & relative specular R, T & A
- absolute & relative diffuse R, T & A
- uncertainties ( $2\sigma$ ):
  - specular:  $\leq 0.2\%$
  - diffuse: 1.5 - 3%
  - larger for angle dependent structure
- can measure R of accessories samples
- can sort out scatter from total R & T



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# Comparison of PAN-MA/SWNT, PAN-MA/CB and SWNT films

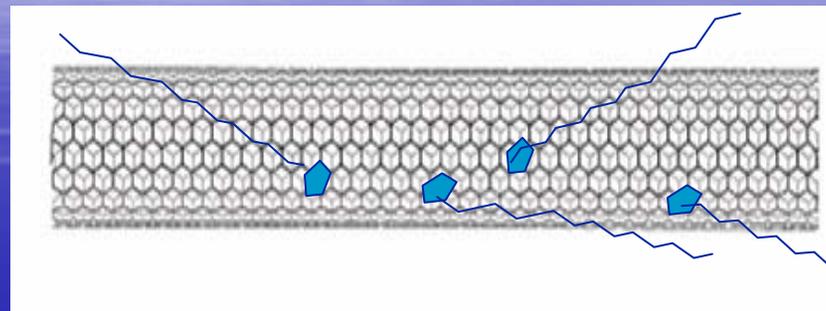
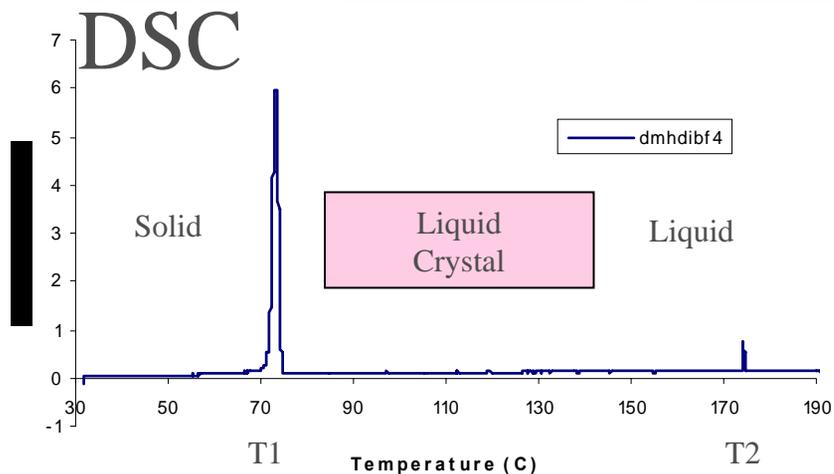


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Surface roughness measurement  
(root-mean-square deviation of the  
surface, PSq) of the  
PAN-MA/SWNT, PAN-MA/CB and  
SWNT films with LSCM

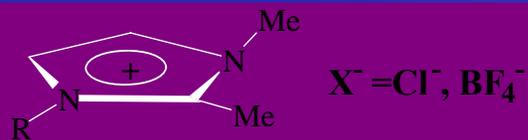
Severine Bellayer

# Nanotube-Ionic liquid solutions

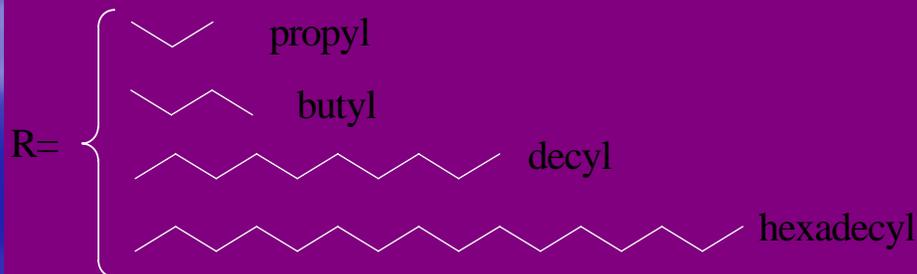


## Imidazolium Surfactant and MWNT

Bellayer, S. Gilman, J. et al. *Adv. Funct. Mater.* 2005

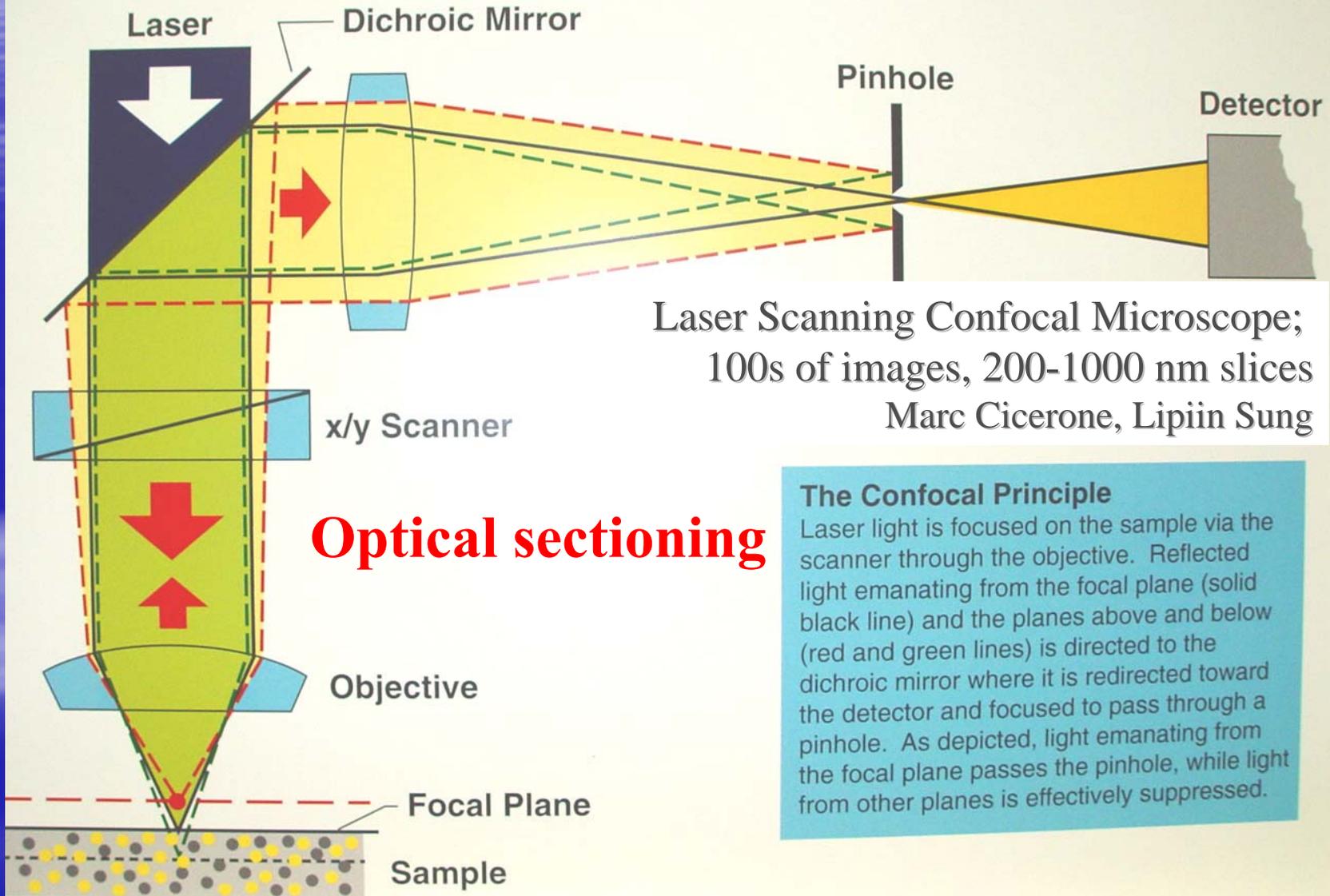


## Dimethyl alkyl imidazolium salts



# Confocal Characterization of Nanocomposites

## Laser Scanning Confocal Microscope

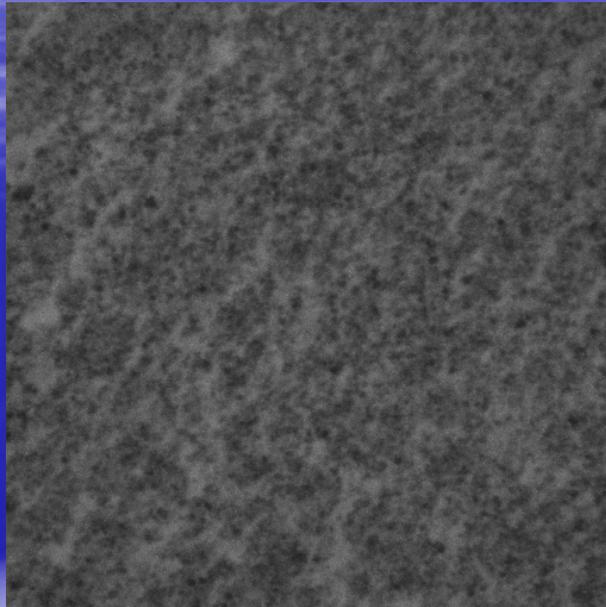


Laser Scanning Confocal Microscope;  
100s of images, 200-1000 nm slices  
Marc Cicerone, Lipiin Sung

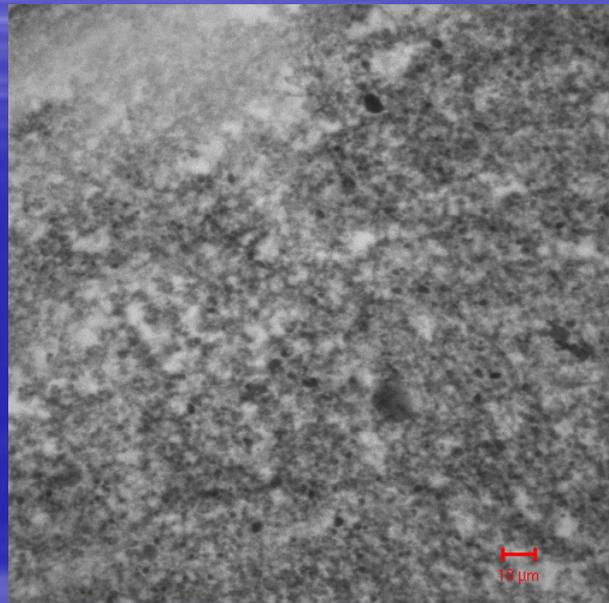
### The Confocal Principle

Laser light is focused on the sample via the scanner through the objective. Reflected light emanating from the focal plane (solid black line) and the planes above and below (red and green lines) is directed to the dichroic mirror where it is redirected toward the detector and focused to pass through a pinhole. As depicted, light emanating from the focal plane passes the pinhole, while light from other planes is effectively suppressed.

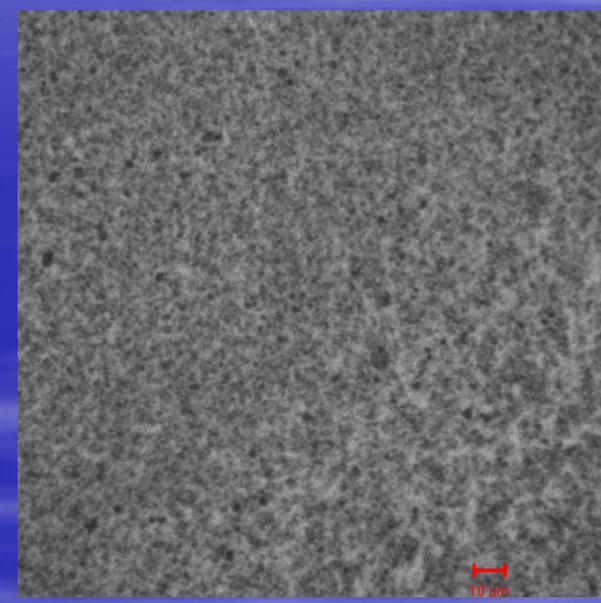
# Dispersion of MWNT in Ionic Liquids



BMICl +  
MWNT



BMICl +  
MWNT  
modified w/DMHDI BF4

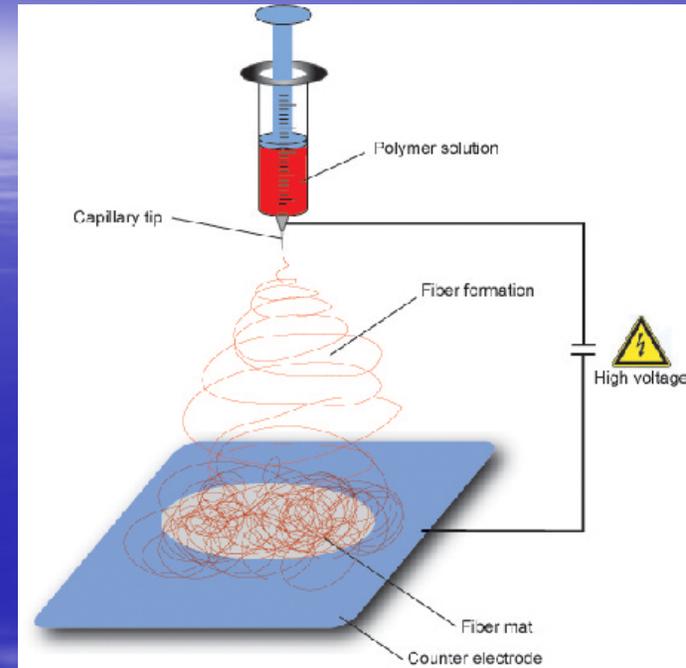
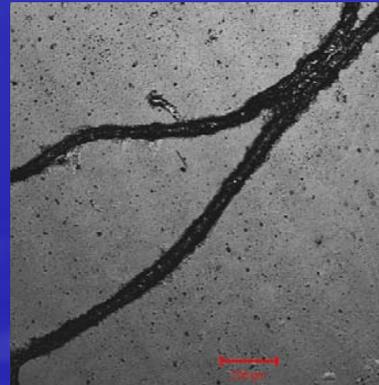


BMICl +  
MWNT  
modified w/EMICl

Fluorescent dye : Rhodamine B, processing: 2 min sonication

# Electrospinning

- MWNT/Imidazolium/Silk fibers



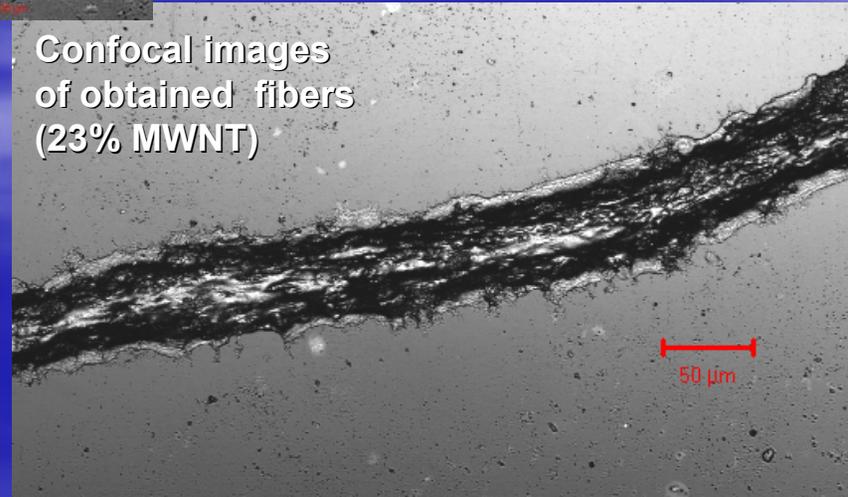
Electrospinning system

[www.chemie.uni-marburg.de](http://www.chemie.uni-marburg.de) JH Wendorff

Goal

Production of fiber with high loading of MWNT

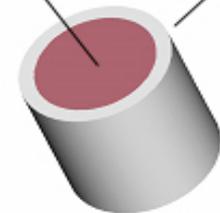
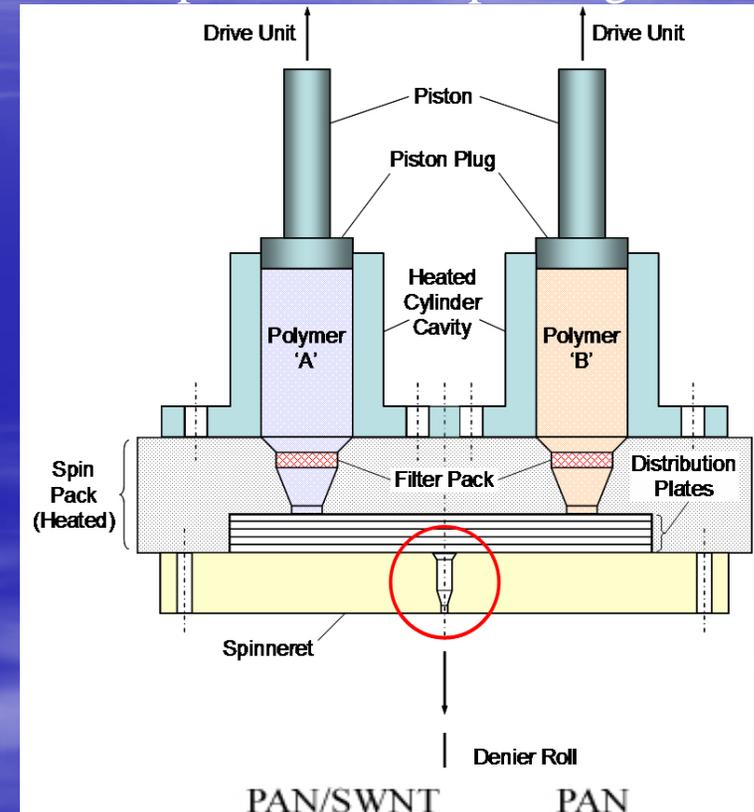
Confocal images  
of obtained fibers  
(23% MWNT)



# Plans

- Develop electro-spinning methods to prepare CNT /polymer fabrics with high loadings
  - MWNT / VGCNF in ionic liquids
- Collaborate with Satish Kumar @ Georgia Tech
  - electro-spinning
  - Bi-component fiber spinning

Bi-component fiber spinning unit



# Plans

- Thermal Characterization
  - Evaluate Pitch-based Carbon Fiber (PBCF)
  - Collaborate with Satish Kumar @ Georgia Tech
    - PBCF fabric

- Pitch-based Carbon Fiber
  - Thermal Conductivity
    - 500-1000 W/mK

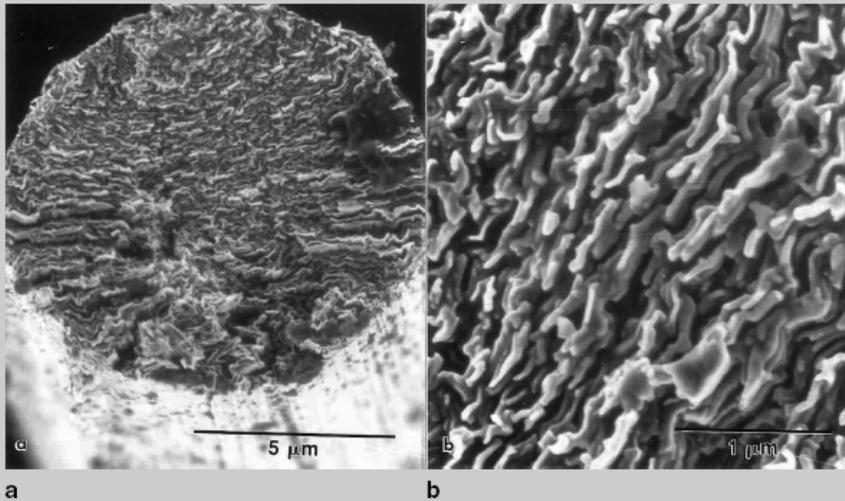


Figure 7. A scanning-electron micrograph of pitch-based P-100 fiber at (a) low and (b) high magnification.<sup>52</sup>

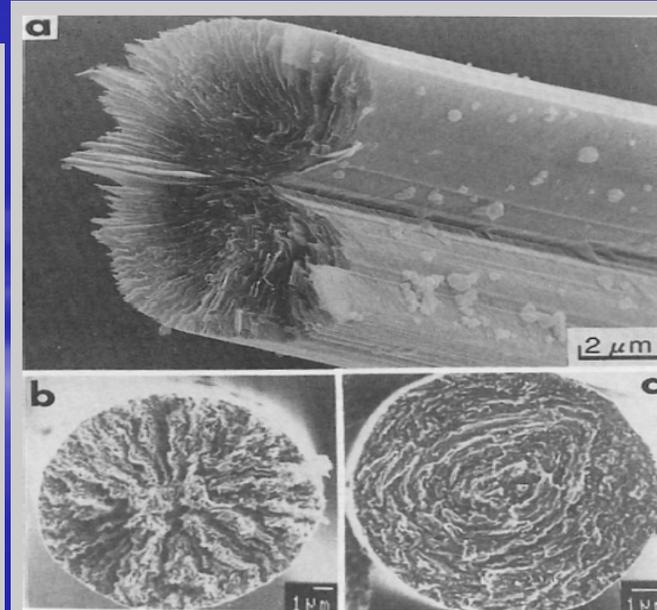
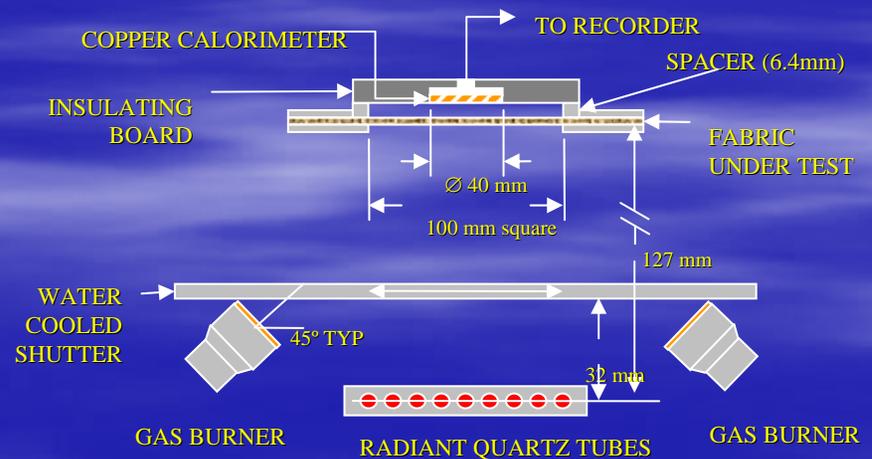


Figure 5. Typical transverse microstructures for pitch-based carbon fibers showing (a) radial with wedge, (b) radial, and (c) concentric microstructure.<sup>24</sup>

# Plans

- Thermal Characterization of Fabrics
  - Radiative Apparatus
  - ISO 17492
  - Mannequin



# Mattress Barrier Fabric

## TB603 Burner Exposure

Tom Ohlemiller



## Barrier Materials

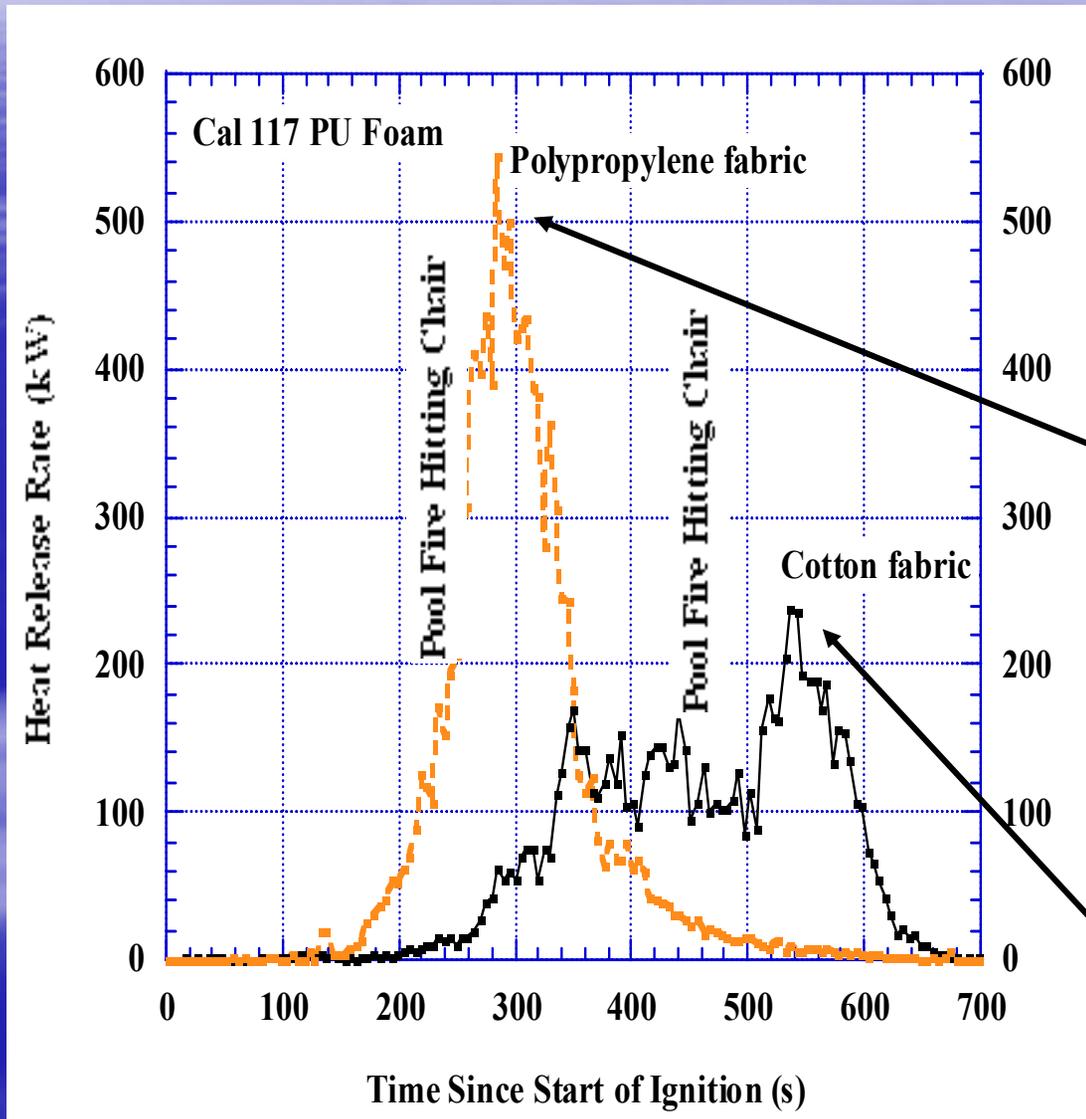
Aramid

Novoloid

PVA-PVC

Modacrylic/polyester blend

# Fire Barrier Fabric



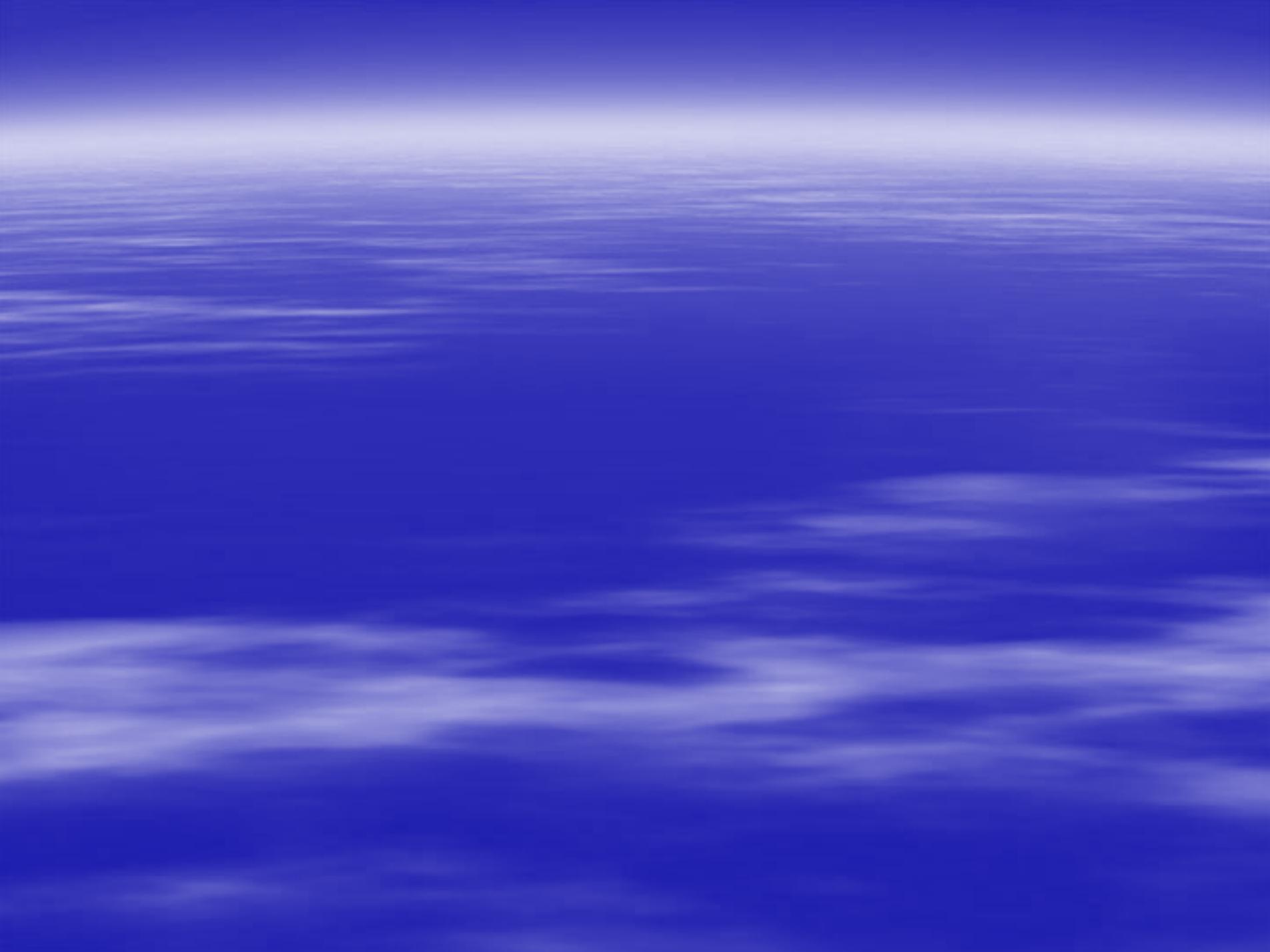
# Conclusions

- ❑ Pure carbon fabrics offer potential thermal protection
- ❑ Electro-spinning Imidazolium-CNT solutions offers potential for CNT rich fabric
- ❑ Bi-component fiber spinning offers potential for CNT rich fabric
- ❑ Confocal, IR Camera and Radiative Apparatus offer rapid characterization
- ❑ CNT/polymer fabrics hold promise as barrier fabrics

# Research Team

**Severine Bellayer,**  
**Randy Shields, Richard Harris, Marc Nyden,-NIST**  
**Paul Maupin –DOE**  
**Paul C. Trulove and Doug Fox –Naval Academy**  
**Hugh DeLong -Air Force Office of Scientific Research**  
**Satish Kumar – Georgia Tech**

**New Postdoc:**  
**Sameer S. Rahatekar**  
**Department of Materials Science and Metallurgy**  
**University of Cambridge**  
**Cambridge, UK**



# Radiative Gasification Apparatus

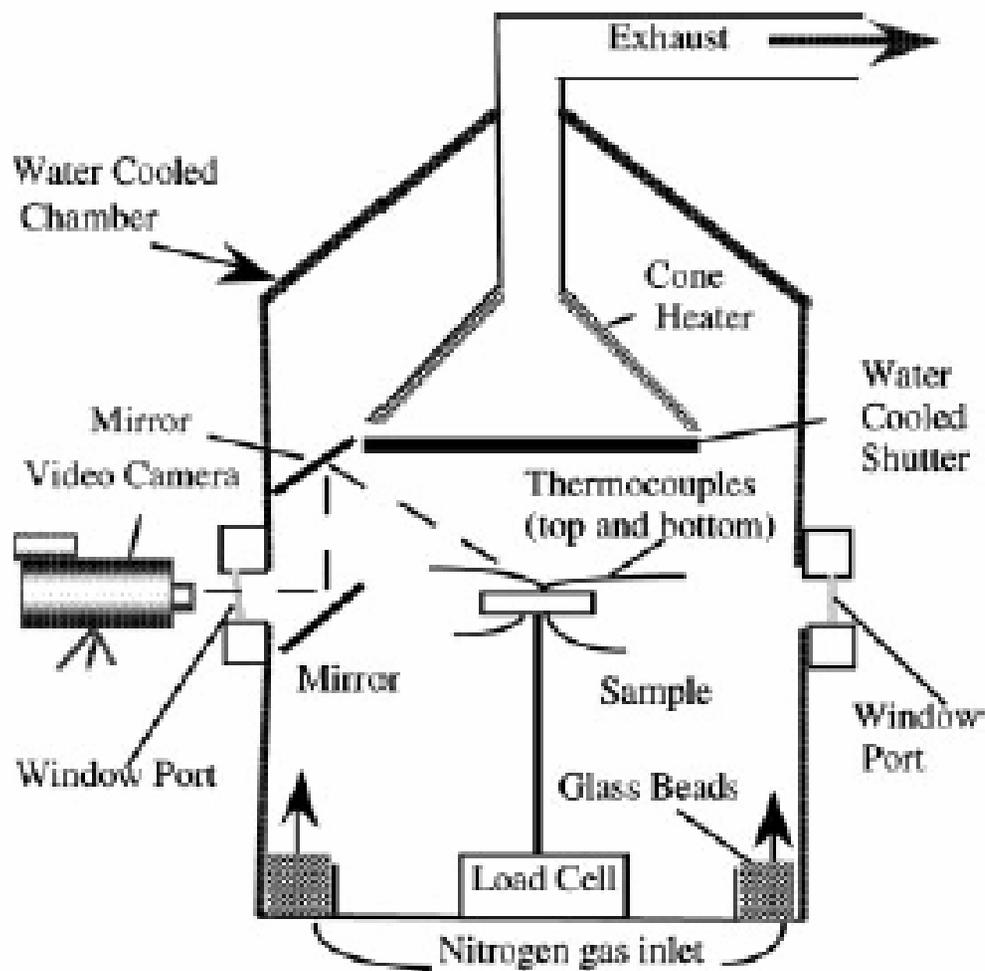


Figure 1. Schematic of gasification device.



# PROTECTION FROM HEAT & FLAME

NFPA 1971 (TPP = Thermal Protective Performance rating)

ISO 17492 (TTI = Thermal-protection index)

ASTM D4108 (withdrawn)

## Heat Sources:

NFPA 1971 - Two burners plus radiant quartz tubes

ISO 17492 - Two burners plus radiant quartz tubes

ASTM D4108 - One burner, no radiant quartz tubes

## Calibration:

NFPA 1971 - Radiant quartz tubes =  $12 \text{ KW} / \text{m}^2$ , balance from burners

ISO 17492 - Equally balanced convection and radiation

ASTM D4108 - Single adjustment of flame

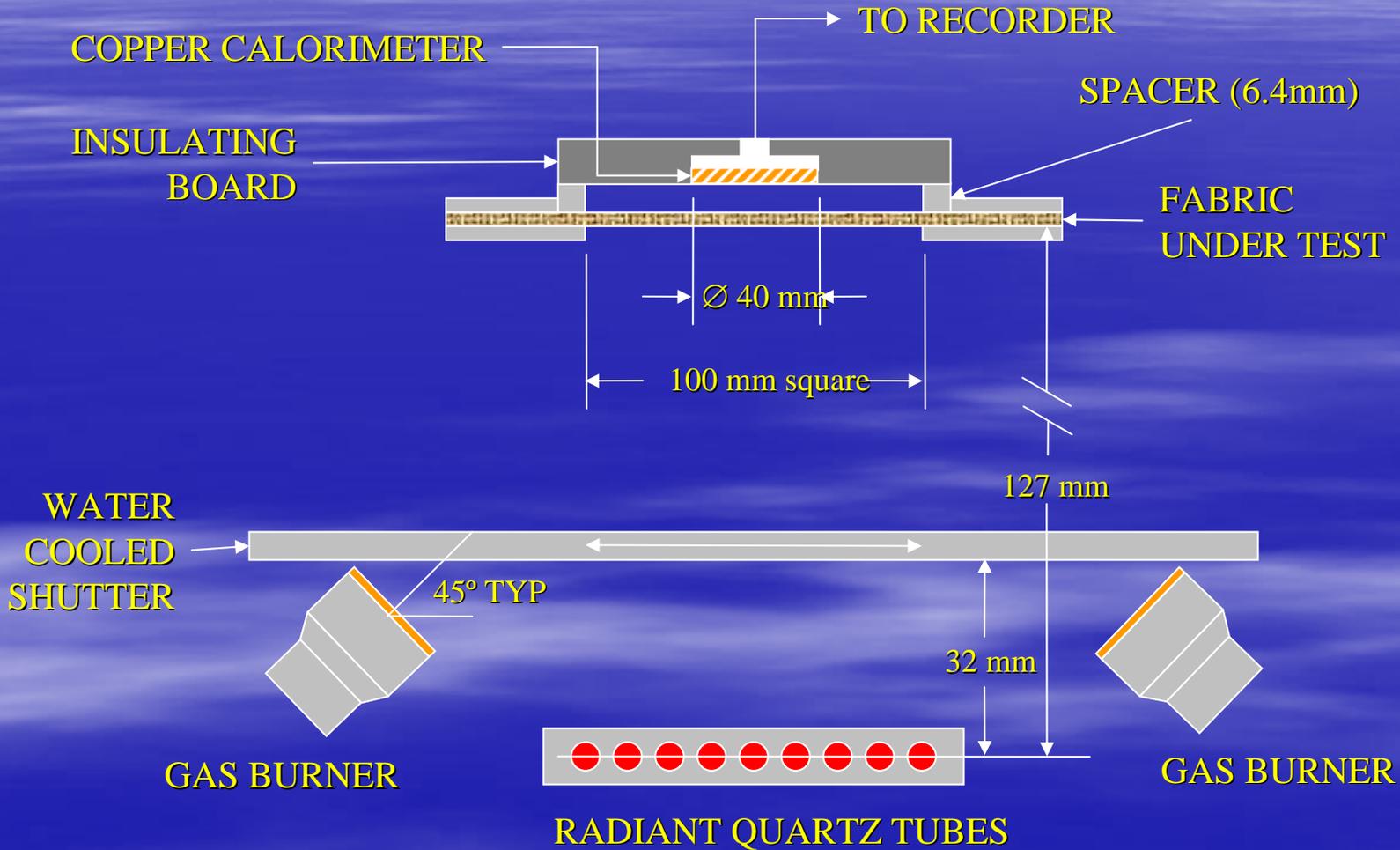
## Spacer blocks:

NFPA 1971 - No spacer block

ISO 17492 - Spacer for single-layered, no spacer for multi-layered

ASTM D4108 - Spacer for single-layered, no spacer for multi-layered

# Test apparatus, single layered fabric: ISO 17492



# Thermal Protective Performance (TPP) rating

